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PRODUCTION OF Ti_3SiC_2 -BASED MATERIALS BY SHS FORCED COMPACTION OF LAYERED COMPOSITE Ti–SiC

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A new method of synthesizing ceramic composite materials based on Ti_3SiC_2 using non-powder reaction compositions of titanium and silicon carbide was developed. A ceramic composite with a Ti_3SiC_2 – TiSi_2 matrix reinforced with SiC particles was obtained by SHS forced compaction of a multilayer packet of regularly packed layers of titanium foil and polymer film filled with silicon carbide particles. The particulars of the phase composition and microstructure of the material obtained were investigated.

Key words: Ti_3SiC_2 , self-propagating high-temperature synthesis, SHS compaction.

The innovational potential of Ti_3SiC_2 -based ceramic materials is due to their unique combination of properties: high resistance to cracking, insensitivity to thermal shock, and good mechanical workability. These properties are mainly due to layered character of the Ti_3SiC_2 crystalline structure, expressed as a relatively weak bond between carbide layers $[\text{Ti}_3\text{C}_2]_{\infty\infty}$ and the layers of silicon atoms $[\text{Si}]_{\infty\infty}$ separating them. Such a nano-laminated structure makes it possible for Ti_3SiC_2 grains to deform strongly under a load, localizing the mechanical damage to the material thereby preventing macroscopic fracture [1]. A diagram of the crystalline structure of Ti_3SiC_2 and the microstructure of its grains is presented in Fig. 1.

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The high cost and technical complexity of the existing methods of production is holding back the adoption of Ti_3SiC_2 -based materials in industry: hot isostatic pressing [2 – 5], spark plasma sintering [6, 7], chemical precipitation from the gas phase [8, 9], and so on. Finding technologically efficient and cost-effective methods of production is among the most pressing problems at the present stage of research. A promising approach to solving this problem could be the application of self-propagating high-temperature synthesis (SHS) using layered reaction compositions consisting of a packet of regularly packed reagents in the form of films and foils. For the production of large articles such a technology greatly simplifies the procedure for forming articles, which gives tangible advantages over powder methods.

This article presents the results of laboratory experiments on obtaining Ti_3SiC_2 – TiSi_2 –SiC ceramics by SHS forced compaction of layered reaction composition Ti–SiC.

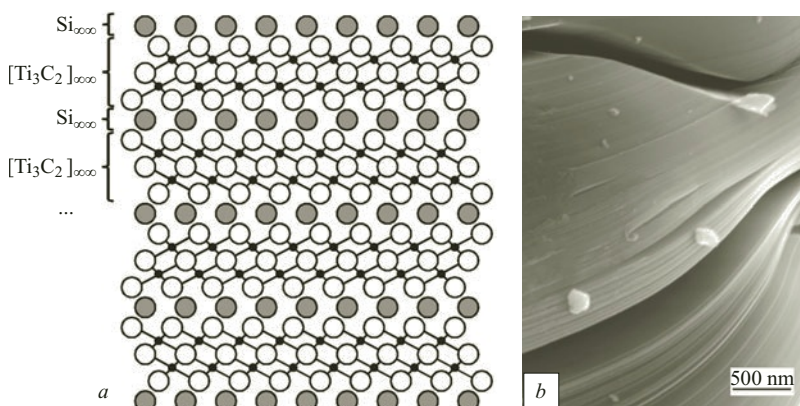


Fig. 1. Diagram of the crystal lattice (a) and microstructure of Ti_3SiC_2 grains (b).

The following were used as sources of the reagents: 55 μm thick VT1-0 titanium foil; 135 μm thick polymer film fabricated from polyvinyl alcohol (PVA); and, filler consisting of isometric silicon carbide particles with average size 20 μm and 70 wt.% SiC. The samples formed by multilayer packing of the reagents, as shown in Fig. 2, were subjected to vacuum heat-treatment under a mechanical load 200 kN/m^2 . The temperature regime included two isothermal sections: 1) 30 min, 400°C to remove PVA and 2) 15 min, 1340°C to perform the synthesis.

The composition of the products of synthesis was monitored by x-ray phase analysis using a Shimadzu XRD-6000 diffractometer (CuK_α radiation, Ni filter). The POWDER CELL computer code was used to analyze the diffraction patterns [10]. A LOMO Polam P-312 microscope was used to study the microstructure.

It was determined in the course of the experiments that the SHS reaction is initiated during the heat-treatment of the layered composite Ti–SiC at temperature 1320°C and that this reaction is accompanied by self-heating of the sample, which can be observed visually. As Fig. 3 shows, x-ray phase analysis of the products of synthesis shows the presence of 65, 14, and 18 vol.% Ti_3SiC_2 , TiSi_2 , and SiC, respectively, as well as traces of TiC and $\text{Ti}_5\text{Si}_3\text{C}_x$. The lineal shrinkage in the direction of the applied load reaches 25 – 30%, while the planar dimensions of the sample remain practically unchanged during heat-treatment. The apparent density of the material obtained is 3.36 g/cm^3 , which is 80% of the theoretical value. The relatively high residual porosity is probably due to fact that the load applied during SHS compaction was too small for effective compaction.

Figure 4 compares fragments of the microstructure of the sample and its pre-treatment structure (Fig. 4a). It is important to note that a large part of the synthesized composite is characterized by a uniform distribution of the SiC particles and isolated pores in a Ti_3SiC_2 – TiSi_2 matrix (Fig. 4c). Indications of the initially present micro-laminations are seen in the sample only fragmentally, predominately in the periph-

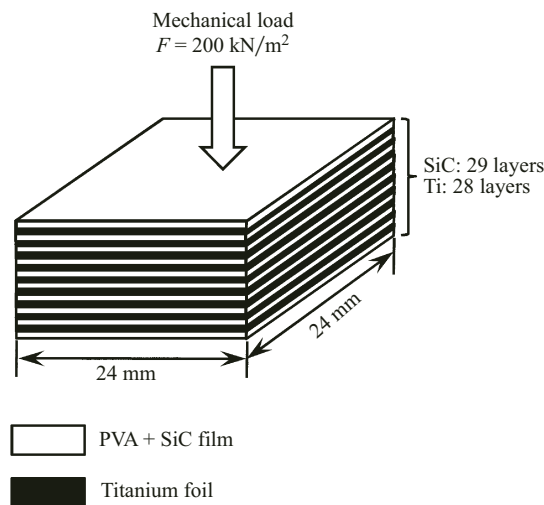


Fig. 2. Diagram of the packing of the initial reagents.

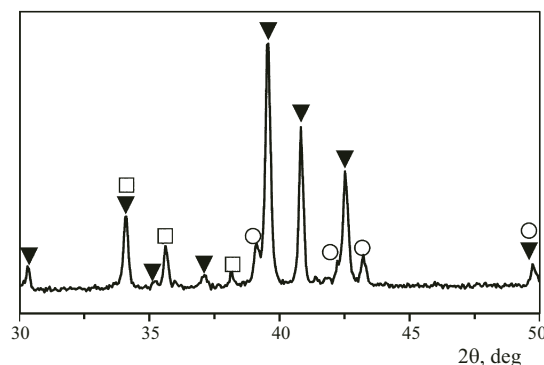
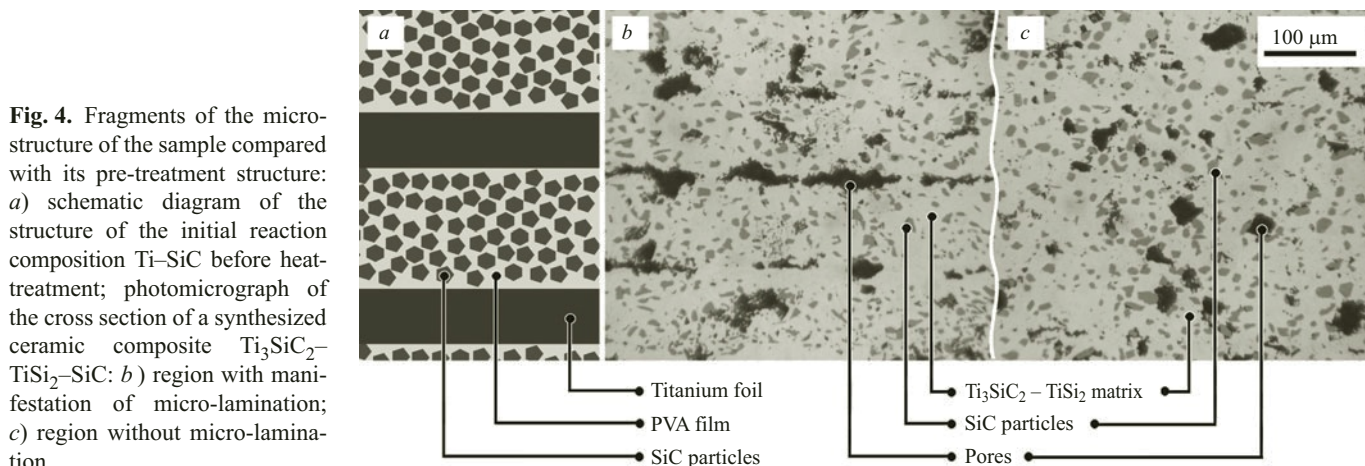


Fig. 3. Diffraction pattern of the products of synthesis: ▼) Ti_3SiC_2 ; ○) TiSi_2 ; □) SiC.

ral regions, as clusters of oriented pores localized at the location of the initial packing of the titanium layers (Fig. 4b).

Comparing the results obtained and the published data [11, 12] it can be concluded that on the whole the mechanism



of phase formation in the experimental layered system Ti–SiC is no different from the mechanism operating in powder reaction mixtures of the elements and binary compounds of the system Ti–Si–C. At temperature 1320°C the interaction of the initial reagents, which below 1320°C occurred by diffusion, switches into the SHS interaction regime. The combustion stage is accompanied by the formation of a titanium-silicon-carbon melt followed by crystallization of Ti_3SiC_2 and TiSi_2 as the melt cools. The material is compacted under the applied mechanical load, and in the process the unreacted SiC is uniformly distributed in the Ti_3SiC_2 – TiSi_2 matrix. According to the diagram of phase equilibria of the system Ti–Si–C [13] the attained composition of the products is close to the equilibrium at the heat-treatment temperature. On the whole the process can be represented by the overall reaction



In summary, it has been shown experimentally that ceramic composite materials with a matrix based on Ti_3SiC_2 can be obtained, in principle, by SHS compaction of layered non-powder compositions Ti–SiC under a mechanical load. The technical-economic advantages of the method described are low consumption of energy and time as well as good technological workability for the production of large-size articles.

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